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(54) Title: METHOD FOR THE MANUFACTURE OF LYOCELL FIBRE

(57) Abstract

Never-dried lyocell fibres can be treated with an aqueous solution which contains an alkali metal hydroxide and from 1 to 50 grams per litre of an alkali metal silicate (calculated as anhydrous sodium metasilicate). The presence of the silicate serves to minimise the potential for damage to the fibre during this alkaline treatment step, particularly when the solution contains from 5 to 15 percent by weight sodium hydroxide.

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METHOD FOR THE MANUFACTURE OF LYOCELL FIBRE

Field of the invention

This invention relates to processes for the manufacture of lyocell fibre which include the step of contacting the fibre in never-dried state with an aqueous solution of an alkali metal hydroxide.

Background art

Lyocell fibres are known, and their manufacture is described for example in US-A-4,416,698, the contents of which are incorporated herein by way of reference. Cellulose is dissolved in a solvent containing a tertiary amine N-oxide (which may also be called for brevity an amine oxide), for example N-methylmorpholine N-oxide (NMMO). The solvent generally also contains a proportion of a non-solvent for cellulose, for example water. The resulting solution is extruded through a suitable die to produce fibres, which are coagulated, washed in water to remove the solvent and dried. This process of extrusion and coagulation is referred to as "solvent spinning", and the cellulose fibre produced thereby is referred to as "solvent-spun" cellulose fibre or as lyocell fibre. It is also known that cellulose fibres can be made by extrusion of a solution of a cellulose derivative into a coagulating and regenerating bath. One example of such a process is the viscose process, in which the cellulose derivative is cellulose xanthate. Solvent spinning has a number of advantages over other known processes for the manufacture of elongate cellulose members, such as the viscose process, for example reduced environmental emissions.

Lyocell fibres are known to be prone to fibrillation. Fibrillation is a phenomenon which in the main occurs when lyocell fibres are subjected to mechanical forces during wet-processing, and it results in the partial detachment of fine longitudinal fibrils from the fibres. Fibrillation is in general considered to be undesirable in textile end-uses, and efforts have been made to reduce or eliminate fibrillation tendency by chemical aftertreatments, such as

those described in US-A-5,310,424, or by suitable choice of spinning parameters, as described for example in WO-A-95/02082.

International Patent Application PCT\GB96\03160 published as WO97/23668 describes a process for the manufacture of an extruded lyocell article such as fibre which includes the characterising step of applying to the reconstituted but never-dried lyocell article an aqueous solution of an alkali metal hydroxide containing from 0.20 to 3.85 percent by weight hydroxide ions. An aqueous solution containing from 0.5 to 9 percent by weight sodium hydroxide may be used. The aqueous solution of alkali metal hydroxide may conveniently be applied to the fibre from a circulating bath, the residence time of the reconstituted article therein conveniently being in the range from 20 to 90 seconds. This process is said to enable the manufacture of lyocell articles having increased dyeability, increased whiteness, reduced yellowness and/or increased absorbency.

Unpublished International Patent Application PCT\GB97\1459 filed on 29th May 1997 describes a process for the manufacture of lyocell fibre which includes the characterising step of applying to the reconstituted but never-dried fibre for 20 seconds or more an aqueous liquor which comprises from 10 to 18 percent by weight sodium hydroxide. This process is said to enable the manufacture of lyocell fibre with controlled, in particular reduced, fibrillation tendency.

It is known that cellulose exhibits a swelling maximum in aqueous sodium hydroxide at a sodium hydroxide concentration of about 10 percent by weight. It is also known that the degree of swelling of cellulose in aqueous sodium hydroxide generally increases as the temperature is reduced. The present inventors have found that treatment of never-dried lyocell fibres with aqueous solutions containing from 5 to 15 percent by weight sodium hydroxide can damage

the fibre, particularly at low temperatures, unless great care is taken. Such damage may manifest itself as loss of tensile properties, loss of lustre, loss of weight by dissolution or by detachment of fibrils, or in severe cases destruction of the fibre. This can render the processes described in the aforementioned British patent applications difficult of control. The present invention seeks to overcome this difficulty.

Disclosure of invention

Under the present invention lyocell fibre is manufactured by a method which includes the step of contacting the fibre in never-dried state with an aqueous solution of an alkali metal hydroxide which additionally comprises from 1 to 50, preferably from 5 to 20, grams per litre of an alkali metal silicate (calculated as anhydrous sodium metasilicate, Na_2SiO_3).

According to the invention there is provided a method for the manufacture of lyocell fibre, including the steps in sequential order of:

(1) extruding a solution of cellulose in an organic solvent through a die, thereby producing an elongate form;

(2) passing the elongate form through at least one water-containing bath to remove the organic solvent from the elongate form, thereby producing a reconstituted cellulosic member;

(3) applying to the reconstituted cellulosic member an aqueous solution of an alkali metal hydroxide;

(4) washing the reconstituted cellulosic member to remove alkali metal hydroxide therefrom; and

(5) drying the reconstituted cellulosic member, thereby forming the lyocell fibre,

characterised in that the aqueous solution of an alkali metal hydroxide additionally comprises an alkali metal silicate at a concentration in the range from 1 to 50, preferably from 5 to 20, grams per litre (calculated as anhydrous sodium metasilicate Na_2SiO_3). The fibre is dried

for the first time in step (5), so that the reconstituted cellulosic member may alternatively be referred to as never-dried fibre.

The method of the invention is applicable to lyocell fibre in the form of continuous filament yarn, tow or staple fibre. The titre of the lyocell fibre may be in the range from 0.5 to 10 decitex. When the method of the invention is practised on lyocell fibre in the form of continuous filament yarn or tow, the fibre is preferably maintained in relaxed state during the characterising step of the invention.

The alkali metal hydroxide is preferably sodium hydroxide, although other compounds such as potassium hydroxide may alternatively be used. The aqueous solution of alkali metal hydroxide may comprise from 5 to 15, preferably from 8 to 13, percent by weight sodium hydroxide.

The alkali metal in the alkali metal silicate is preferably sodium. The material known as waterglass is a convenient commercial form of sodium silicate for making up an aqueous solution for use in the invention.

The temperature of the aqueous solution of alkali metal hydroxide is preferably in the range from 0 to 60°C, more preferably from 15 to 30°C.

The aqueous solution of alkali metal hydroxide may be applied to the fibre by any convenient conventional means, for example using a circulating bath, wicking roller or spray. When a circulating bath is used, the residence time of the fibre therein may conveniently be in the range from 5 to 120 seconds.

After the alkali treatment step, alkali metal hydroxide is washed from the fibre. In one embodiment of the invention, the fibre is washed with hot water, preferably

followed by a sour wash with dilute aqueous acid so that the fibre pH is below 7. In another embodiment of the invention, the fibre is washed with an aqueous acid solution. The acid may be a mineral acid such as hydrochloric acid or sulphuric acid, the concentration thereof in the aqueous acid solution being in the range from 0.1 to 20, preferably from 1 to 15, percent by volume, or it may be an organic acid such as acetic acid, the concentration thereof in the aqueous acid solution being in the range from 25 to 75, preferably from 40 to 60, percent by volume. The washing liquor may be applied to the fibre by any convenient means, for example using a circulating bath, wicking roller or spray. When an aqueous acid solution is used, the residence time of the fibre in such a circulating bath may conveniently be in the range from 5 to 120 seconds.

The method of the invention has the advantage that it can be carried out on conventional equipment.

Fibrillation was induced and degree of fibrillation assessed using Test Method 1.

Test Method 1

Approximately 0.05 g of dry fibre is cut to 10 mm lengths and placed in a laboratory blender. 400 ml of tap water is added and the mixture is blended for 30 seconds - 3 minutes. The actual length of time required depends on the blade being used and is chosen to give a fibrillation index of 6.5 - 8.0 for a sample of standard commercial lyocell fibre (Tencel (Trademark), Courtaulds Fibres (Holdings) Limited, Grimsby, U.K.).

The blended fibres are collected and a few are placed on a microscope slide. Another few fibres are placed on a second slide. The fibres are compared under a microscope to a set of standard graded photographs of fibrillated lyocell fibre. Three sets of five readings are taken on each slide

and averaged to give the Fibrillation Index (F.I.). An F.I. of zero corresponds to zero fibrillation.

The invention is illustrated by the following Examples, in which parts and proportions are by weight unless otherwise specified.

Example 1

A solution of cellulose (15%) in NMMO (75%) and water (10%) was extruded through a spinnerette into an aqueous coagulating bath to produce 1.7 dtex filaments. After washing with water to remove NMMO, the filaments were treated in relaxed state with an aqueous solution comprising 11% w/v sodium hydroxide and varying amounts of sodium silicate for 30 seconds at 25°C, washed in relaxed state with 15% v/v aqueous sulphuric acid solution for 30 seconds, rinsed and dried. Further experimental details and results are given in Table 1:

Table 1

Sodium silicate g/l	Titre dtex	Tenacity cN/tex	Extension %	F.I.
Untreated control	1.84	37.3	15.2	6.2
0	1.87	29.1	13.7	2.1
10	1.79	35.2	15.7	2.3
20	1.73	39.4	15.8	2.6
30	1.75	38.0	16.2	3.1
50	1.80	38.3	15.2	3.7

The solutions were made up using waterglass nominally containing 244 g/l sodium metasilicate.

Fibre treated according to the method of the invention exhibited a combination of good tensile properties and a low tendency to fibrillation, as well as good lustre and freedom from damage resulting from alkali treatment.

CLAIMS

1. A method for the manufacture of lyocell fibre, including the steps in sequential order of:

(1) extruding a solution of cellulose in an organic solvent through a die, thereby producing an elongate form;

(2) passing the elongate form through at least one water-containing bath to remove the organic solvent from the elongate form, thereby producing a reconstituted cellulosic member;

(3) applying to the reconstituted cellulosic member an aqueous solution of an alkali metal hydroxide;

(4) washing the reconstituted cellulosic member to remove alkali metal hydroxide therefrom; and

(5) drying the reconstituted cellulosic member, thereby forming the lyocell fibre;

characterised in that the aqueous solution of an alkali metal hydroxide additionally comprises an alkali metal silicate at a concentration in the range from 1 to 50, preferably from 5 to 20, grams per litre (calculated as anhydrous sodium metasilicate).

2. A method according to claim 1, further characterised in that the alkali metal hydroxide is sodium hydroxide.

3. A method according to claim 2, further characterised in that the concentration of sodium hydroxide in the aqueous solution is in the range from 5 to 15, preferably from 8 to 13, percent by weight.

4. A method according to any one of the preceding claims, further characterised in that the alkali metal in the alkali metal silicate is sodium.

5. A method according to any one of the preceding claims, further characterised in that the temperature of the

aqueous solution is in the range from 0 to 60°C, preferably from 15 to 30°C.

6. A method according to any one of the preceding claims, further characterised in that the reconstituted member in step (3) is maintained in relaxed state in the form of continuous filament yarn or tow.

7. A method according to any one of the preceding claims, further characterised in that the washing step (4) involves washing firstly with hot water and secondly with dilute aqueous acid whereby the pH of the lyocell fibre is below 7.

8. A method according to any one of claims 1 to 6, further characterised in that the washing step (4) involves washing with an aqueous solution containing from 0.1 to 20, preferably from 1 to 15, percent by volume of an acid selected from the group consisting of hydrochloric acid and sulphuric acid.

9. A method according to any one of claims 1 to 6, further characterised in that the washing step (4) involves washing with an aqueous solution containing from 25 to 75, preferably from 40 to 60, percent by volume of acetic acid.

INTERNATIONAL SEARCH REPORT

Internat Application No
PCT/GB 97/01959

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D01F2/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 D01F C08J D06M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	WO 95 24524 A (COURTAULDS FIBRES HOLDINGS LTD ;TAYLOR JAMES MARTIN (GB)) 14 September 1995 see the whole document	
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☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

...rmation on patent family members

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